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Synthesis and Characterization of Vanadium Oxide Aerogels

by

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SYNTHESIS AND CHARACTERIZATION OF VANADIUM OXIDE AEROGELS

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ABSTRACT

Vanadium pentoxide aerogels were synthesized by supercritical drying with CO₂. The aerogels were prepared using a variety of sol compositions from the system VO(OC₃H₇)₃/H₂O/acetone. The materials were found to be of fairly low density (0.04g/cm³ to 0.1g/cm³) with surface areas in the range of 300 to 400 m²/g. Chemical and structural studies indicate that the aerogels are hydrated oxides of composition V₂O₅ • nH₂O with $n = 2.0$ to 2.2 and possess a fibrous morphology. When partially dehydrated, the vanadate aerogels exhibit electron transport with conductivity and activation energy values comparable to those of xerogels. Electrochemical measurements demonstrate that lithium can be intercalated reversibly into the structure.

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1. INTRODUCTION

Aerogels are high surface area, low density solids prepared by the sol-gel approach followed by supercritical drying of the wet gel [1]. The aerogel field has expanded substantially in recent years and applied to a number of metal oxide and mixed metal oxide compositions. For many years, aerogels found uses as catalysts because the high surface area led to more active sites for gas/solid interactions[2]. In more recent times, other applications have emerged for these materials including thermal and acoustic insulation, Cerenkov detectors and ultrasonic sensor systems [1,3].

The present paper considers the synthesis and properties of vanadium pentoxide aerogels. The sol-gel chemistry of vanadium pentoxide is quite well known and provides a strong basis for this work [4]. There is considerable flexibility in the synthesis of vanadate sols and gels; a widely used approach is through the ion exchange of sodium metavanadate solutions [5]. The V_2O_5 colloids which form from the sol-gel process exhibit a fibrous morphology which, upon and gelation and drying (to form a xerogel), produce some form of preferred stacking with two-dimensional order [6]. This anisotropy is particularly strong in xerogel thin films. The gels are hydrous oxides of composition $V_2O_5 \cdot nH_2O$ and, when dried under ambient conditions, stable xerogels contain about 1.8 H_2O per V_2O_5 . Studies have shown that three different types of water molecules are hydrogen bonded to the xerogel matrix and that a significant fraction of the water is located between the oxide layers [4].

One particularly attractive feature of vanadate gels is their use as reversible cathodes in lithium batteries. [6] The Li/V_2O_5 xerogel system possesses a high open circuit potential, a large energy density and has shown the ability to intercalate up to 3 lithium/ V_2O_5 for high capacity. The versatile processing of this material by sol-gel methods is of considerable advantage for device fabrication and solid-state thin film batteries based on sol-gel V_2O_5 cathodes and polymer electrolytes have been demonstrated.[7] With the exception of carbon aerogels [8], the electrochemical properties of aerogels have not been explored. Their fine colloid size, high

surface area and controllable porosity, suggest that these materials are likely to exhibit interesting intercalation as well as capacitance properties.

A previous study detailed the synthesis of V_2O_5 bulk aerogels and coatings based on the supercritical drying of ethanol [9]. In this initial work, only one V_2O_5 sol composition was studied ($[H_2O]/[VO(OC_2H_5)_3] = 15$) for which a low density material composed of a fibrous microstructure was obtained. The materials exhibited a bimodal pore size distribution with surface areas in the range of 140 - 200 m^2/g . A conductivity value at 25°C was reported along with x-ray diffraction patterns. For the latter, it is interesting to mention that because of the elevated temperature required for supercritical drying (255°C), some crystallization peaks were evident in the as-dried aerogel with no evidence of preferred orientation.

In the present paper, a new approach for synthesizing vanadate aerogels is described where supercritical drying of CO_2 is utilized for a wide range of sol compositions. The synthesis conditions are detailed and a number of physical, chemical and electrical properties of the vanadate aerogels are presented. The materials are found to be hydrated oxides with surface areas in the range of 300 - 400 m^2/g . Partially hydrated materials are electronically conducting and lithium ions can be intercalated electrochemically into the structure.

2. EXPERIMENTAL PROCEDURE

2.1. Synthesis method

Monolithic vanadium oxide aerogels were prepared by supercritical drying with liquid CO_2 . The process is shown schematically in Figure 1. The alkoxide precursor used, vanadyl triisopropoxide, $VO(OC_3H_7)_3$ (Gelest, Inc.), was hydrolyzed in acetone and condensed to form a "wet gel." In a typical preparation (e.g., sample number 19) 5.64 cm^3 of an acetone / water solution was added to 0.80 cm^3 of $VO(OC_3H_7)_3$ to achieve the molar ratio of 1/30/15 for the system $VO(OC_3H_7)_3/H_2O/acetone$. The components were stirred vigorously and no catalyst was

added. Prior to mixing, the solutions were maintained in an ice bath. The acetone-water molar ratio was varied extensively and materials were synthesized over a wide composition range.

The wet gels were aged for 1 - 3 days at room temperature in sealed test tubes. Longer aging times in open tubes resulted in denser gels that had better mechanical integrity. After aging, the samples were removed from the test tubes, immersed and then washed several times with anhydrous acetone. In order to ensure complete replacement of the initial water-acetone mixture, eight washings were generally required. The acetone-exchanged gels were then transferred to the pressure vessel (Polaron E3000 Critical Point Dryer) for supercritical extraction by CO₂. A typical procedure involved cooling the chamber to 15°C before filling with liquid CO₂. Repetitive purge-fill cycles were employed to flush acetone from the gel. The temperature was then raised to 42°C while the pressure was increased to 1500 psi. After approximately 5 minutes, the CO₂ was vented slowly. The temperature and pressure conditions are shown in Figure 2. Typical samples were prepared in the form of rods, 1 cm in diameter by 3 cm in length.

2.2. Characterization

The density of the vanadate aerogels was determined by using a pycnometer vial filled with mercury. A Quantachrome Autosorb-1 gas sorption system was used to determine surface area and total pore volume. Nitrogen gas was used as the adsorbate and, prior to the measurements, the samples were powdered and outgassed for 12 hours at 150°C under vacuum. The BET surface area (taken at $P/P_0 = 0.31$) and total pore volume (taken at P/P_0 close to unity) were calculated from 40 point adsorption and desorption isotherms.

Thermogravimetric analysis (TGA) of the vanadate aerogels was obtained using a DuPont 9900 Thermal Analysis System. Sol-gel methods lead to hydrated vanadium oxides (i.e., $V_2O_5 \cdot nH_2O$), thus it is of interest to establish the value of n for the aerogel materials. Several compositions were exposed to laboratory air after synthesis and allowed to stabilize. The

weight loss upon heating was measured from 25 to 500°C at a heating rate of 10°C/min in nitrogen atmosphere.

Information concerning structure and morphology of the aerogels was obtained by x-ray diffraction (XRD), infrared spectroscopy and transmission electron microscopy (TEM). The XRD pattern was similar to that reported by Hirashima et. al. for V_2O_5 xerogels prepared from $VO(OC_2H_5)_3$ [10]. This pattern does not index to the 00 ℓ set of peaks commonly observed for gels prepared from aqueous solution [6]. The interpretation of this pattern will be treated in a future publication. FTIR measurements (Bomem Model BM-100) were made on finely powdered samples placed between two KRS 5 windows. TEM was carried out on a Philips CM30. These samples were prepared by placing finely ground aerogel powders between two holey carbon grids.

Electrical conductivity of the vanadate aerogels was measured between room temperature and 150°C by the complex impedance method. A spring-loaded sample holder was used in which thin brass foil electrodes made contact to gold electrodes which were sputtered onto opposite faces of an aerogel disc. The sample was then placed inside a glass tube so that the measurements were made in a controlled atmosphere. Complex impedance measurements were made over the frequency range 10 Hz to 100 kHz using a lock in analyzer (EG & G Model 5207). In a typical experiment, the sample was heated to 150°C for one hour in flowing argon and measurements were taken upon cooling. The temperature was stabilized for at least 10 minutes prior to taking data at a given temperature.

3. RESULTS

3.1. Aerogel Synthesis

The composition diagram (Figure 3) indicates the range of synthesis conditions explored. The gels in Region I could be prepared readily as monolithic aerogels. The as-prepared gels were deep red in color except for those synthesized at low water concentration

($\text{H}_2\text{O}/\text{VO}(\text{OC}_3\text{H}_7)_3 < 5$) which were orange. After the gels aged for several days, the color changed from red and orange to brown and green, respectively. The materials gelled rapidly (< 3 minutes) with the shortest times (5 sec) generally observed for the samples with the lowest acetone/ $\text{VO}(\text{OC}_3\text{H}_7)_3$ molar ratio. Gelatinous precipitates were obtained with materials prepared in Region II of Figure 3 and thus no aerogels were synthesized using these compositions. The shaded section (highest water content) represents a group of compositions which produced aerogels that were badly deformed.

Table 1 provides selected information for representative vanadate aerogel compositions identified in Figure 3. The table includes details on gelation time, density and shrinkage for samples prepared with different molar ratios of water/ $\text{VO}(\text{OC}_3\text{H}_7)_3$ and acetone/ $\text{VO}(\text{OC}_3\text{H}_7)_3$. The shrinkage occurred prior to supercritical drying, during the aging and washing stages. With few exceptions, the synthesis conditions produce materials which are of quite low density; from 0.04 g/cm^3 to 0.1 g/cm^3 . This corresponds to a solid content between 1% and 3%.

3.2. Characterization

These studies involved the characterization of aerogel morphology and the corresponding chemical and structural properties. A group of representative vanadate aerogel samples were found to possess surface areas in the range of 300 to $400 \text{ m}^2/\text{g}$ and pore volumes of between 1 and $1.5 \text{ cm}^3/\text{g}$ (Table 2). It should be noted that these samples were heated to 150°C for 12 hours prior to the gas adsorption/desorption measurements so that values of as-prepared aerogels may differ somewhat from those obtained here. A separate set of identical aerogel samples were analyzed by TGA (Table 2). The total weight loss upon heating between room temperature and 500°C is in the range of 16.5 to 18%. This suggests that the aerogels are characterized by the composition $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ with $n \approx 2.0$ to 2.2. This is slightly greater than the $\text{V}_2\text{O}_5 \cdot 1.8 \text{ H}_2\text{O}$ identified for vanadate xerogels dried under ambient conditions [4]. The weight change profile (Figure 4) is characterized by a steep loss between room temperature and 100°C , followed by a more gradual weight change to 350°C .

Infrared spectroscopy results for the aerogels also indicate that this material may be described as a hydrated oxide. The infrared absorption spectrum (Figure 5) is similar to that reported for vanadate xerogels prepared from alkoxide precursors as it is dominated by absorption bands due to water molecules (at 3500 cm^{-1} and 1625 cm^{-1}), H_3O^+ at 1735 cm^{-1} and vanadium-oxygen bonds [11]. The latter is indicated by both the strong absorption at $\approx 1000\text{ cm}^{-1}$ (V = O double bonds) and the broad bands from the V-O-V oxide network observed at lower frequency [12]. The fact that no bands from V - OC vibrations are evident indicates that all the alkoxy groups have been removed upon hydrolysis.

TEM investigations of the vanadate aerogels indicate the fibrous nature of their morphology (Figure 6). The ribbon-like structure of these fibers is similar to that reported previously for vanadate xerogels [4, 6] and aerogels [9]. It is also apparent from the TEM that the individual ribbons are composed of narrower sub-units. In bulk aerogel materials, it would seem that the fibers condense and aggregate in a random fashion as is suggested by Figure 6. However, the presence of peaks in the XRD pattern suggest that the ribbons exhibit some sort of preferred arrangement.

3.3. Transport Properties

Electrical conductivity measurements for the vanadate aerogels were carried out on samples which had been heat treated for one hour at 150°C in flowing Argon. According to the TGA results, this heat treatment removed approximately 75% of the water contained in the gel so that the aerogel corresponded to a composition $\text{V}_2\text{O}_5 \cdot 0.4\text{ H}_2\text{O}$. At this level of dehydration, the conductivity of V_2O_5 xerogels is electronic in nature, occurring by an electron hopping mechanism [4, 11]. The aerogels behave similarly. The electronic nature of the conductivity is evident by the fact that the sample impedance was independent of frequency and that the phase angle was close to zero. The conductivity - temperature relationship for the V_2O_5 aerogels (Figure 7) indicates an Arrhenius type behavior of the form

$$\sigma = \sigma_0 \exp (-E/kT)$$

where σ_0 is the pre-exponential constant and E is the activation energy for conduction. The conductivity at room temperature is $\approx 1 \times 10^{-4}$ S/cm and the activation energy is ≈ 0.25 eV.

4. DISCUSSION

The previous work on vanadate aerogels involved supercritical drying of alcohol and was based on the system $\text{VO}(\text{OC}_2\text{H}_5)_3/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$. Only one sol composition from this system was investigated [9]. The present aerogel synthesis approach is substantially different as it is based on supercritical drying by CO_2 . The ethanol-based system is not appropriate for the CO_2 process, however, the flexibility of sol-gel chemistry enables one to create another sol system which is compatible. Acetone was chosen as a common solvent because of its miscibility with water and liquid CO_2 , and a convenient alkoxide precursor, $\text{VO}(\text{OC}_3\text{H}_7)_3$, was utilized. A wide range of compositions in the system water/acetone/ $\text{VO}(\text{OC}_3\text{H}_7)_3$ were prepared as aerogels as shown in Figure 3 and Table 1. Although the properties of the resulting aerogels have not been studied comprehensively, the initial results suggest that some of the properties are relatively independent of the sol composition. For example, the values obtained for density, surface area and water content exhibit relatively little variation (see Tables 1 and 2). Perhaps this behavior is influenced by aerogel morphology, because the TEM results indicate that a similar fibrous morphology is observed for all samples throughout Region I (Figure 6).

The hydrated nature of the V_2O_5 aerogels is readily apparent from infrared spectroscopy (Figure 5). TGA results suggest that the amount of water in the $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ aerogels is $n = 2.0$ to 2.2. This value is slightly greater than the 1.8 H_2O per V_2O_5 reported for aqueous derived xerogels dried under ambient conditions [4]. The greater value for n , however, is consistent with the basal plane distance, d , determined from XRD of aerogels. Previous work has shown that the basal plane distance is sensitive to the amount of water (i.e, the value of n) in the $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ gels [4]. In vanadate xerogels, $d = 11.6 \text{ \AA}$ for $n = 1.8$, while in the aerogels, $d = 12.7 \text{ \AA}$. Another

interesting feature in the TGA is the distinct curvature in the weight loss characteristics in the region between 100 and 125°C (Figure 4). The steeper gradient below 100°C is likely to correspond to the departure of weakly bound water molecules. In xerogels, the hydration state at 120°C is $n = 0.5$ as water molecules are considered to be chemically bound in cavities between oxide layers [4]. For the aerogels, the change in curvature in this temperature range suggests a similar interpretation, although the values for n are somewhat greater than $n = 0.5$.

The electrical properties of the aerogels are similar to those reported for xerogels prepared from alkoxide precursors [11]. ESR measurements indicate that the vanadate aerogels are mixed valence materials and that the fraction of reduced vanadium ($[V^{4+}]/[V^{4+}] + [V^{5+}]$) is approximately 10%. Thus, electronic conduction in the aerogel arises from electron hopping between V^{4+} and V^{5+} . Additional studies are necessary to establish whether transport follows a small polaron mechanism. It is interesting to note that the activation energy, E , is in excellent agreement with that reported for the alkoxy-derived xerogels while the conductivity at 25°C, $\approx 1 \times 10^{-4}$ S/cm, is a factor of ten lower. In view of the much lower solids content of the aerogels, it appears that the conductivity scales with the solid fraction of the vanadate material. In contrast, the room temperature conductivity of the CO_2 processed aerogel (Figure 7) is about ten times greater than the value reported for the alcohol-based aerogel [9].

The interest in V_2O_5 as a cathode material for secondary lithium batteries has led us to investigate the electrochemical properties of vanadate aerogels. In recent years, there have been a number of investigations of thin film vanadate xerogels and promising results have been obtained [4, 6, 7]. The extremely high surface area of aerogels offers the possibility of creating electrode materials where surface conduction dominates and rapid intercalation prevails in small dimensions.

The initial electrochemical experiments with vanadate aerogels were carried out using an electrolyte of 1 M $LiClO_4$ in propylene carbonate. Lithium was used as both the counter and reference electrodes. The working electrode consisted of the V_2O_5 aerogel mixed with carbon black and a binder and then pressed into nickel mesh. Cyclic voltammograms indicated that the

electrode not only exhibited lithium intercalation reactions but also showed capacitive behavior. Chronopotentiometry experiments performed at constant current density ($j = 10 \mu\text{A}/\text{cm}^2$) demonstrated that the aerogel electrode can be cycled through standard charge-discharge operations.

5. CONCLUSIONS

Vanadium oxide aerogels were synthesized by a new approach using supercritical drying with CO_2 . Aerogels were prepared using a variety of sol compositions from the system $\text{VO}(\text{OC}_3\text{H}_7)_3/\text{H}_2\text{O}/\text{acetone}$. The resulting materials were found to be of fairly low density ($0.04 \text{ g}/\text{cm}^3$ to $0.1 \text{ g}/\text{cm}^3$) with surface areas in the range of 300 to $400 \text{ m}^2/\text{g}$. The chemical, structural and electrical properties of the vanadate aerogels exhibit similarities to those of aqueous and alkoxy-derived xerogels. Chemically, the aerogel materials may be described as hydrated oxides of composition $\text{V}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ with $n = 2.0$ to 2.2 . TEM studies indicate that the aerogels possess a fibrous morphology. When partially dehydrated, the aerogels exhibit electron transport with conductivity and activation energy values comparable to those of the xerogels. Initial results indicate that the aerogels are electrochemically active and capable of carrying out lithium intercalation reactions.

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Table 1. Vanadate Aerogel Compositions

Sample	H ₂ O/VO(OC ₃ H ₇) ₃	Acetone/VO(OC ₃ H ₇) ₃	Gel time (sec)	Density (g/cm ³)	Shrinkage (%)
5	10	25	39	0.120	20
7	10	60	60	0.086	25
10	20	17.5	30	0.074	7
12	20	40	50	0.087	24
14	20	75	110	0.043	15
20	30	25	40	0.098	20
23	30	55	65	0.080	30
25	30	75	78	0.080	30
30	40	25	40	0.087	13
31	40	50	75	0.100	20
32	40	75	110	0.062	25
35	60	40	60	0.063	16
37	60	75	115	0.063	28
39	60	100	290	0.036	20
42	80	50	85	0.054	22
48	75	75	145	0.041	20
49	75	100	175	0.038	20

Table 2. Selected Properties of Vanadate Aerogels

Sample	Surface area (m ² /g)	Total pore volume (cm ³ /g)	Total weight loss (%)
1	340	n/a	n/a
20	300	1.43	17
31	325	1.23	18
37	275	0.96	16.5
38	420	1.05	17.5

FIGURE CAPTIONS

Figure 1. Flowchart showing the synthesis of vanadium oxide aerogels.

Figure 2. Temperature and pressure profile for supercritical drying with CO₂

Figure 3. Molar ratios of the sol compositions used in aerogel synthesis. Gels were readily prepared in Region I.

Figure 4. TGA scan for aerogel sample 38. The sample was heated at a rate of 10°C per minute in a nitrogen atmosphere.

Figure 5. Infrared absorption spectrum for sample 48.

Figure 6. Transmission electron micrograph of vanadate aerogels;
upper photo: sample 44; lower photo: sample 49

Figure 7. Conductivity - temperature plot for vanadate aerogel. The sample was heated to 150°C and measurements were made upon cooling in flowing argon.

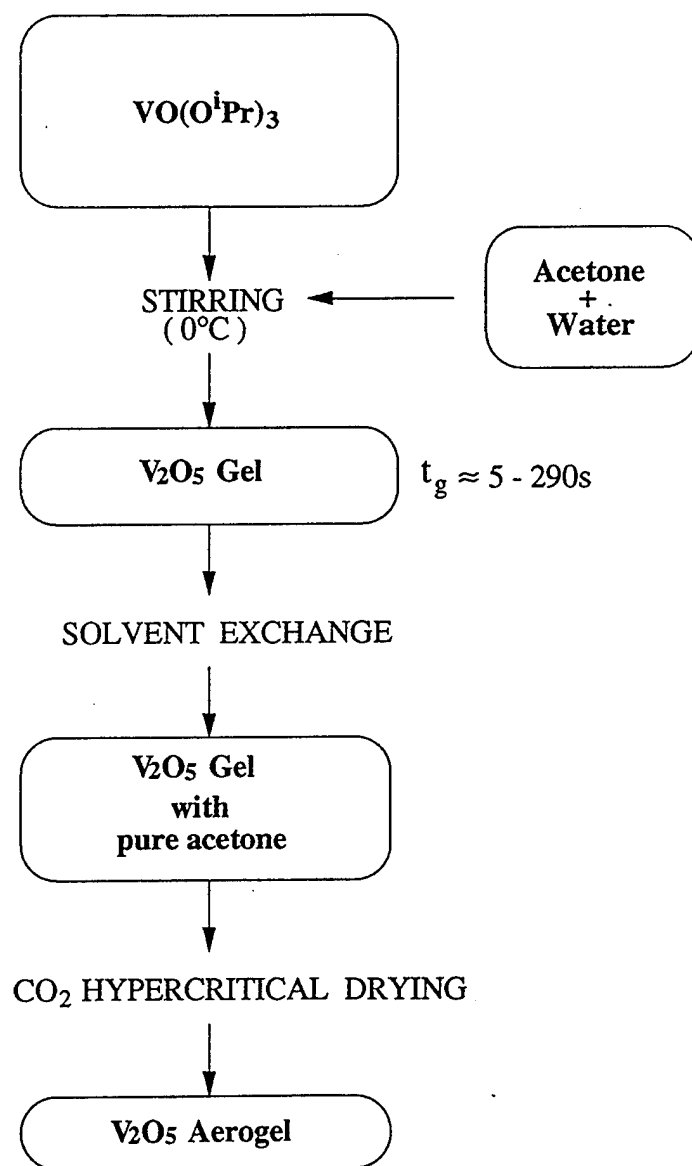


Figure 1. Flowchart showing the synthesis of vanadium oxide aerogels.

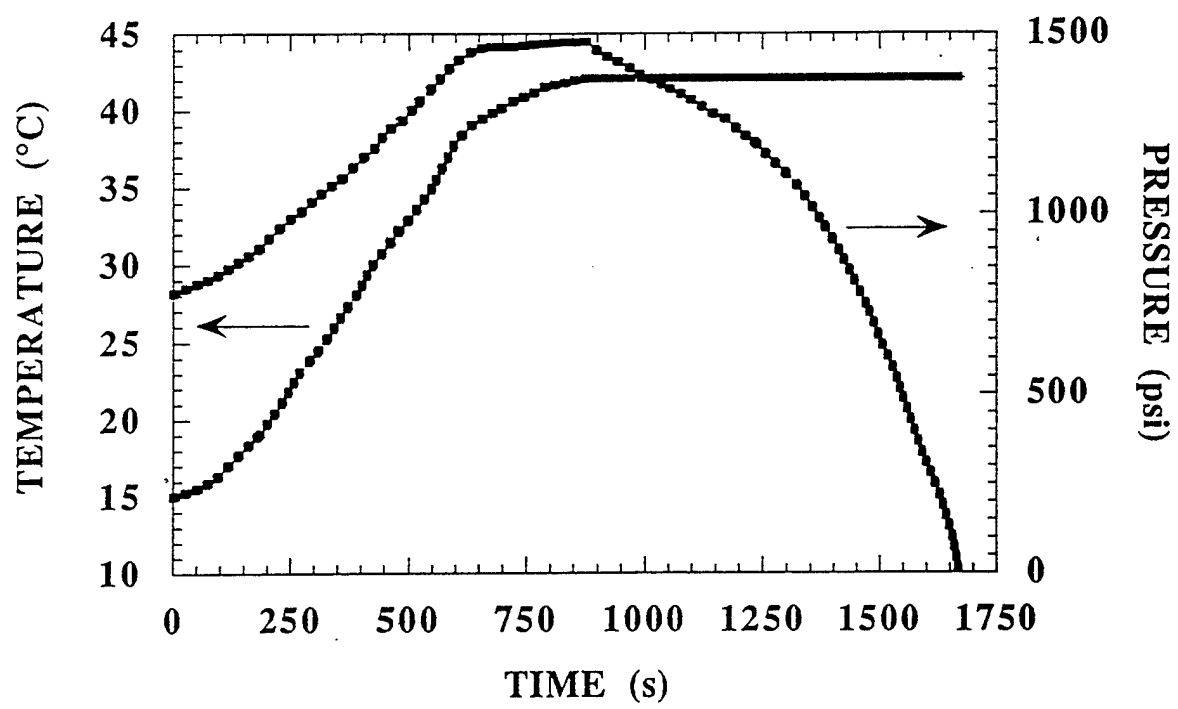


Figure 2. Temperature and pressure profile for supercritical drying with CO₂

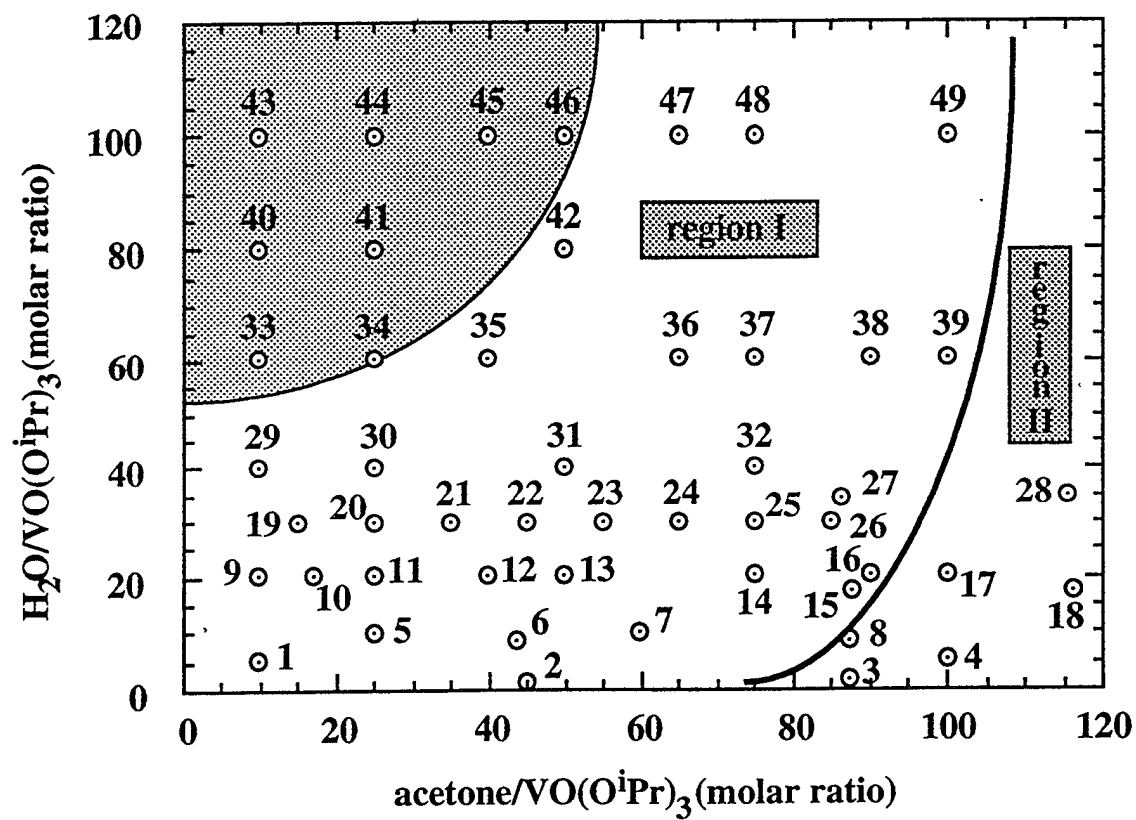


Figure 3. Molar ratios of the sol compositions used in aerogel synthesis. Gels were readily prepared in Region I.

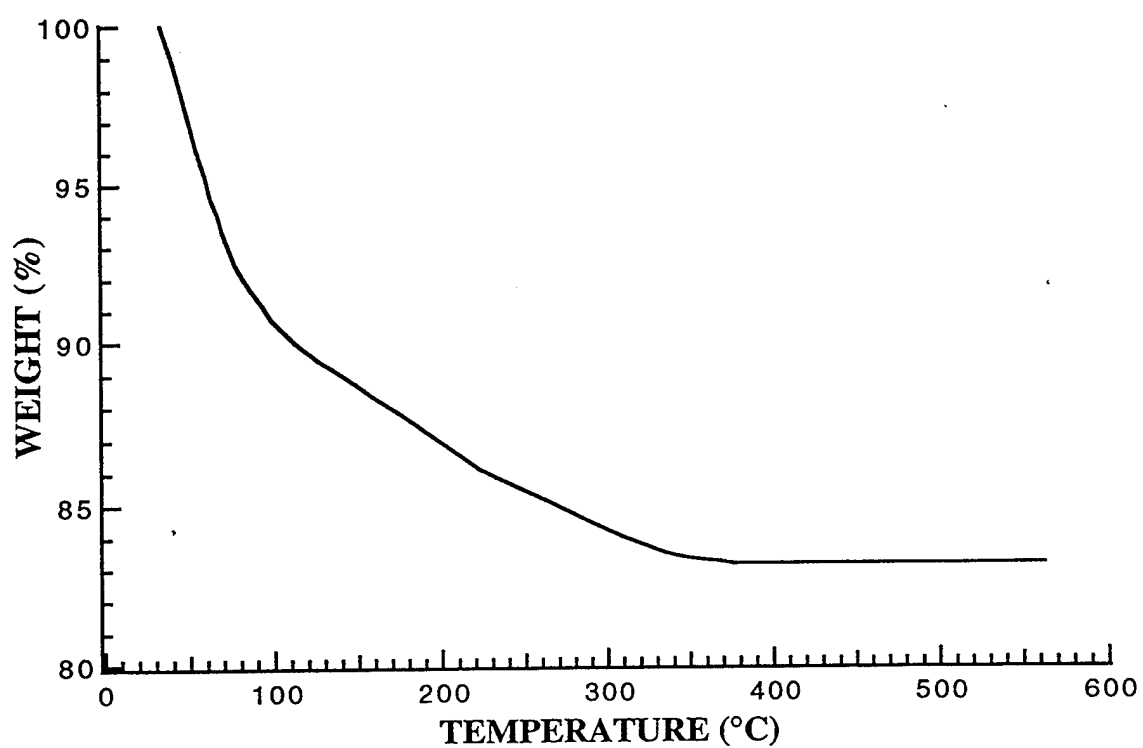


Figure 4. TGA scan for aerogel sample 38. The sample was heated at a rate of 10°C per minute in a nitrogen atmosphere.

Sample 48 cm
figure

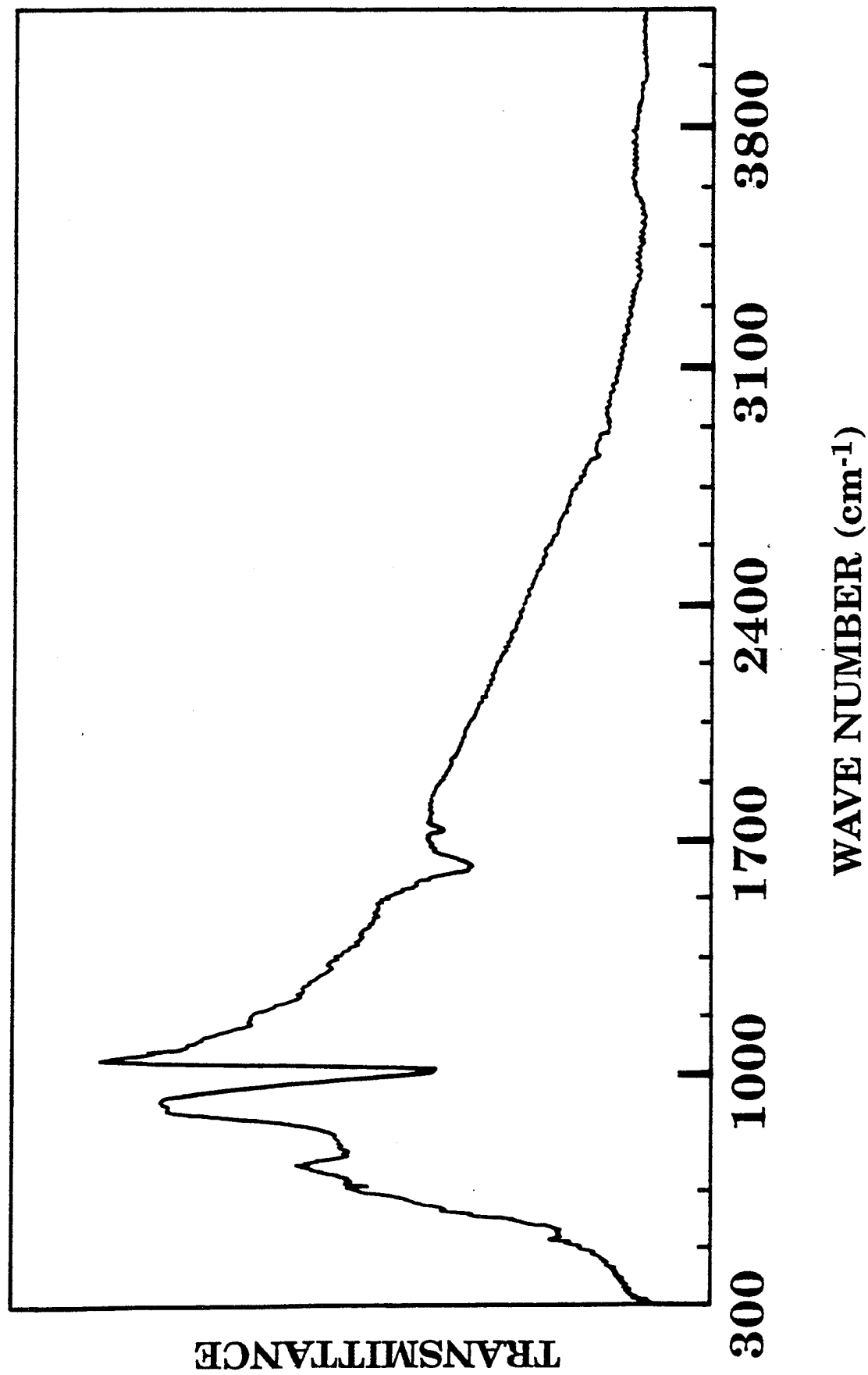


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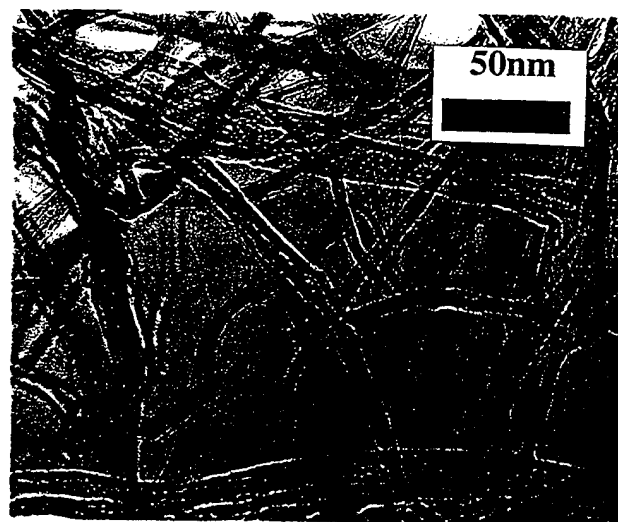
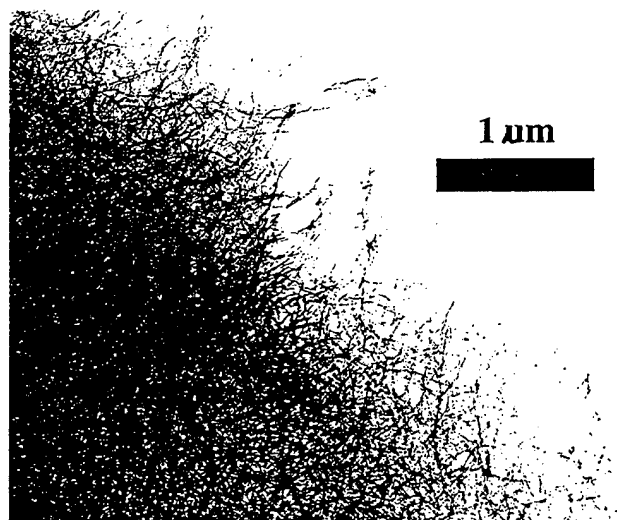


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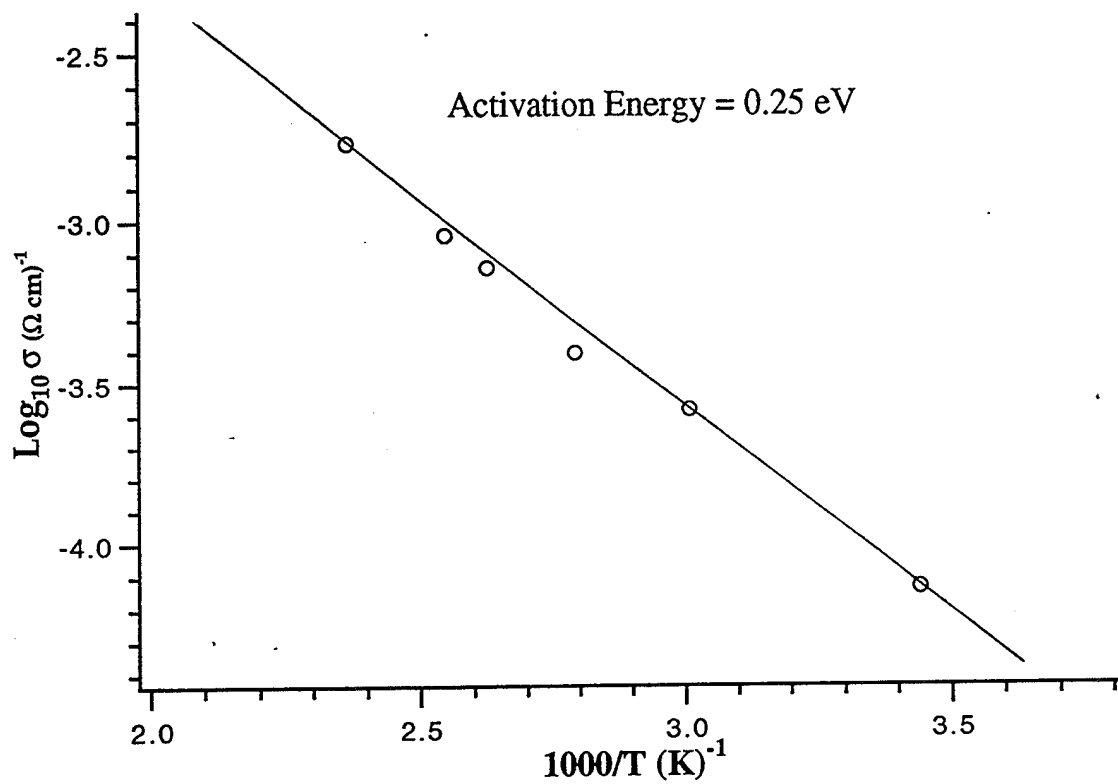


Figure 7. Conductivity - temperature plot for vanadate aerogel. The sample was heated to 150°C and measurements were made upon cooling in flowing argon.

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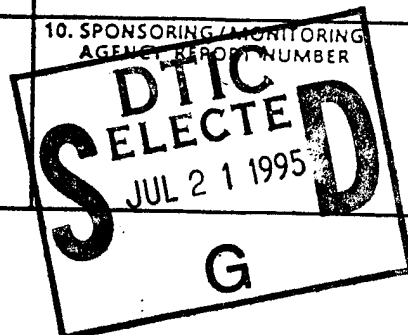
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